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IS 10461-1 (1994): Resistance to Inter-Granular Corrosion of Austenitic Stainless Steels - Method for Determination, Part 1: Corrosion Test in Nitric Acid Medium by Measurement of Loss in Mass (Huey Test) [MTD 24: Corrosion Protection]

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(पहला पुनरीक्षण)

Indian Standard

RESISTANCE TO INTER-GRANULAR CORROSION OF AUSTENITIC STAINLESS STEELS — METHOD FOR DETERMINATION

PART 1 CORROSION TEST IN NITRIC ACID MEDIUM BY MEASUREMENT OF LOSS IN MASS (HUEY TEST)

(First Revision)

UDC 669·14·018·8-194·56 : 620·193

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NEW DELHI 110002

May 1994 **Price Group 2**

Corrosion Protection Sectional Committee, MTD 24

FOREWORD

This Indian Standard (Part 1) (First Revision) was adopted by Bureau of Indian Standards, after the draft finalized by the Corrosion Protection Sectional Committee had been approved by Metallurgical Engineering Division Council.

This standard was first published in 1983 based on ISO 3651/1-1976 (E) Austenitic Stainless Steels—Determination of resistance to intergranular corrosion: Part 1 Corrosion test in nitric acid medium by measurement of loss in mass (Huey Test), issued by the International Organization for Standardization.

While reviewing this standard in the light of experience gained during these years, the committee decided to revise this standard bringing it in line with present practices being followed in the country.

In this revision Figure for Apparatus has been added.

Intergranular corrosion denotes deterioration of metals by means of preferential attacking of the grain boundries. For austenitic stainless steels, which may be subject to such as attack when they are kept at a temperature between about 430 to 820°C, it is associated with the precipitation of chromium rich carbides or intermeallic compounds, such as sigma phase at grain boundries. The heat cycle, which may provoke sensitization to intergranular corrosion, may occur during hot-forming process like forging and rolling, as the result of incorrect solution treatment or during a welding operation.

This standard (Part 1) has been prepared to provide a uniform and rationalized method of determining the susceptibility of austenitic stainless steels to intergranular corrosion.

This standard is issued in two parts, Part 2 of this standard deals with corrosion test in a sulphuric acid/copper sulphate medium in the presence of copper turnings (Monypenny Strauss Test).

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

RESISTANCE TO INTER-GRANULAR CORROSION OF AUSTENITIC STAINLESS STEELS — METHOD FOR DETERMINATION

PART 1 CORROSION TEST IN NITRIC ACID MEDIUM BY MEASUREMENT OF LOSS IN MASS (HUEY TEST)

(First Revision)

1 SCOPE

1.1 This standard describes a procedure for determination of the resistance to intergranular corrosion of austenitic stainless steels in a nitric acid medium by measurement of the loss in mass (Huey Test).

1.2 This method is applicable to austenitic stainless steels supplied in the form of castings, rolled or forged products and tubes including weld metals and intended to be used in an oxidising media (for example, relatively concentrated nitric acid).

1.2.1 The presence or absence of intergranular corrosion in these tests is not necessarily a measure of the performance of the material in other corrosive media. The tests do not provide a basis for predicting the resistance to other forms of corrosion such as general corrosion, pitting or stress corrosion cracking.

2 PURPOSE OF THE TEST

2.1 The test indicates susceptibility to intergranular attack associated with the precipitation of chromium rich carbides or intermetallic compounds such as sigma phase at the grain boundary. Some type of inclusions (active inclusions) are also susceptible to attack in this test.

2.2 This test may be used to evaluate any of the following three factors;

a) the effectiveness of the final treatment when the material is tested in the as delivered condition without further test heat treatment sensitization;

b) the effectiveness of the added stabilizing elements, or low carbon content when the material is tested in the solution heat treated and subsequently sensitized condition; and

c) the influence of welding and associated post fabrication/post weld heat treatments.

3 HEAT TREATMENT FOR SENSITIZATION

In order to verify the effectiveness of the added stabilizing elements or low carbon content to resisting intergranular corrosion, it is necessary to carry out a heat treatment for sensitization. This treatment is usually obtained by maintaining a test pieces for 1 hour at a temperature of 650-675°C followed by rapid cooling to room temperature in air or water unless other specific heating or cooling rates are stipulated. For low carbon variety of stainless steels, the most commonly used heat treatment is heating at 675°C for one hour followed by rapid cooling in air or water. This heat treatment results into appreciably high precipitation of carbides and also simulates to a certain extent microstructure which is obtained after welding.

4 CORROSION TEST

4.1 Principle

A test piece, prepared as specified in **4.4.2**, is weighed; then immersed in a boiling solution of nitric acid for 5 periods, each of 48 hour using fresh solutions for each period. The criterion for evaluating the test is loss in mass determined by weighing after each period.

4.2 Corrosive Solution

The corrosive solution is an aqueous solution of 65 ± 2 percent (m/m) reagent grade nitric acid (r.d. 1.40).

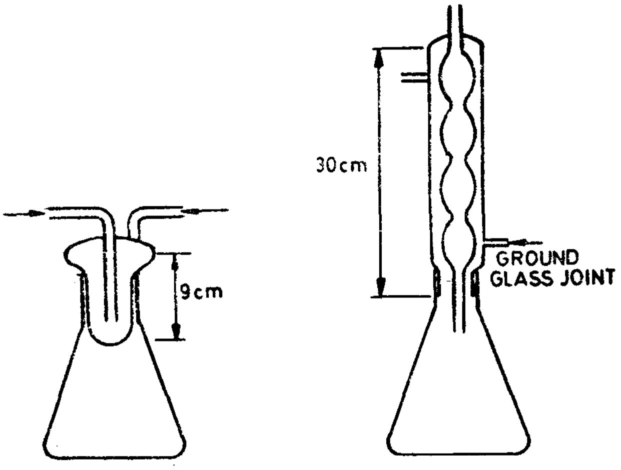
NOTE — The nitric acid conform to the specifications for analytical reagent chemical and shall have the following residual contents:

Fixed residue	< 50 mg/kg
Pb	< 5 mg/kg
Fe	< 2 mg/kg
Mn	Negative test
As	< 0.05 mg/kg
Cl^-	< 1 mg/kg
SO^{4-2}	< 10 mg/kg
PO^{4-3}	< 2 mg/kg
F^-	< 1 mg/kg

4.3 Apparatus

4.3.1 *Conical Flask*

Of capacity at least 1 litre, fitted with a "cold finger" immersion condenser. Other types of condenser, such as Allihn condenser with at least four balbs may be used. The corrosion rates depends on the type of condenser used (*see* Fig. 1). By means of a paper indicator, it shall be checked that no acid fumes are given off during the test. For comparative measurements, the apparatus shall remain identical for all the tests.



COLD FINGER (WIDE MOUTH 6 cm) ALLIHN 45/50 JOINT

ONE LITRE ERLENMEYER FLASKS

FIG. 1 TYPES OF CONDENSERS

4.3.2 *Support for the Test Piece*

Generally made of glass

4.3.3 *Heating Device*

To keep the solution boiling.

4.4 Test Piece

4.4.1 *Dimensions*

The test piece taken from the product shall have its largest dimension located in the direction of working. The test piece dimension shall be determined as a function of the available weighing facilities and the volume of solution to be used. The test piece length shall, however, be at least equal to twice its width, and the total surface area of the section, perpendicular to the direction of working or the fibres, shall be less than 50 percent of the total surface area of the test piece. In comparative tests, the ratio of the total surface area to the total surface area of the sections shall be kept constant. The welded specimens should be cut in such a way that more than 13 mm width of base metal is included in either side of the weld.

4.4.2 *Preparation*

Depending on the purpose of the test (*see* **2.2**), the test piece, either with or without sensitization treatment, shall be prepared as specified in either **4.4.2.1** or **4.4.2.2**. Unless stated on the order to the contrary, the method of preparation shall be left to the discretion of the manufacturer.

4.4.2.1 *Mechanical preparation*

The test piece shall be descaled mechanically by polishing on all surfaces, including the edges, with grade 120 abrasive paper or cloth,

4.4.2.2 *Chemical preparation*

The test piece shall be descaled, without any previous mechanical treatment, in a solution consisting of 50 volumes of hydrochloric acid (r.d 1.19), 5 volumes of nitric acid (r.d 1.40) and 50 volumes of water at 50 to 60°C.

4.4.2.3 *Degreasing*

The test piece shall then be degreased, before being placed in the corrosive solution, and dried using nonchlorinating reagents such as soap and acetone.

NOTE — The preparation treatment described in **4.4.2.1**, may be supplemented by immersing the specimen in nitric acid for example 20 percent (weight) at 50-60°C for 20 minutes followed by degreasing and drying. In case of small diameter tubes which cannot be conveniently mechanically polished on inside, it is suggested to immerse the specimen in boiling nitric acid (65 percent) for 2-4 hours using the same apparatus as for the actual tests. This treatment removes any surface contamination that may not be possible by regular cleaning and would otherwise result in increase in the apparent weight loss of the specimen during early part of the test.

4.5 Procedure

Use a volume of corrosive solution (*see* **4.2**) of at least 20 ml per square centimetre of surface area of the test piece. In general, only one test piece shall be placed in each conical flask. However, it is possible to test several test pieces at the same time, on condition that they all come from the same grade of steel and are isolated from one another at a distance of at least 5 mm.

4.5.1 Determine the mass of the test piece to an accuracy of 0·001 g as well as its surface area to an accuracy of 5 percent. Then immerse the test piece in the corrosive solution and bring the solution to the boil. Boil for 5 periods of 48 h each, using a fresh solution for each period. After each period, wash the test piece in water, dry and weigh.

NOTE — By special agreement between the interested parties, the test may be limited to 3 periods of 48 h each.

4.5.2 In the case of several test pieces being tested in the same flask, the test shall be completely cancelled if, after a period of time, an exaggerated loss in mass of one of the test piece is noted. The test shall be repeated completely on the same test piece which shall be tested separately after having undergone a fresh surface preparation.

4.6 Expression of Results

4.6.1 The effect of the attack by the nitric solution is measured by the mean loss in mass per period.

4.6.2 The corrosion rate is given either in millimetres per year (mm y^{-1}) by formula (1) or in grams per square metre per hour ($\mathrm{g/m^2h^{-1}}$) by formula (2):

$$\frac{87\,600\ \mathrm{m}}{S \times d \times t}\ldots\ldots\ldots\ldots(1)$$

$$\frac{10\,000\ \mathrm{m}}{S \times t}\ldots\ldots\ldots\ldots(2)$$

t = period of attack, in hours;

s = surface area of the test piece, in square centimetres;

m = mean loss in mass per period, in grams; and

d = density of the test piece (1 $\mathrm{g/cm^3}$).

4.6.3 *Sample Results*

Stainless steels (Type 304 and 304 L) are often used in concentrated nitric acid environments as in the case of the processing of nuclear fuels, wastes, etc. In such cases, mass loss in the Huey test shall not exceed 0·46 mm $\mathrm{a^{-1}}$ (equivalent to 18 mills $\mathrm{a^{-1}}$ i.e. 18 mpy). This may be considered as a general guidance. If the corrosion rate for each period increases over that of the previous period, the material can be considered to be susceptible to intergranular corrosion.

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